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A comparison study on non-thermal plasma-assisted catalytic reduction of NO by C_3H_6 at low temperatures between Ag/USY and Ag/Al_2O_3 catalysts

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Abstract

A comparison study was carried out on non-thermal plasma (NTP)-assisted selective catalytic reduction (SCR) of NO_x by propene over Ag/USY and Ag/Al_2O_3 catalysts. Ag/USY was almost inactive in thermal SCR while it showed obvious activities in NTP-assisted SCR at 100 °C–200 °C. Although the NO_x conversion over Ag/Al_2O_3 was also enhanced at 300 °C–400 °C by the assistance of NTP, it was ineffective below 250 °C. The intermediates over Ag/USY and Ag/Al_2O_3 were investigated using in situ DRIFTS method. It was found that key intermediates in HC-SCR, such as NCO, CN, oxygenates and some N-containing organic species were enriched after the assistance of NTP. The differences in the behaviors of above intermediates were not found between these two kinds of catalysts. However, some evidences suggested that different properties of the absorbed NO_x species resulted in the distinction of SCR reactions over Ag/USY and Ag/Al_2O_3. TPD profiles of Ag/Al_2O_3 showed that nitrates formed over the catalyst were quite stable at low temperatures, which might occupy the active sites and were unfavorable to SCR reactions. The nitrates over Ag/USY were unstable, among which the unidentate nitrate species is probably contributed to the SCR reactions at low temperatures.

Keywords: $DeNO_x$; Selective catalytic reduction; USY; Ag/Al_2O_3; Non-thermal plasma-assisted catalysis; In situ DRIFTS

1. Introduction

To improve the activity at low temperatures is essential for selective catalytic reduction of NO_x by hydrocarbons to practical applications. Using NTP as an aid to HC-SCR has shown its value to be effective for extending the effective low-temperature window of metal oxide catalysts. In NTP-assisted C_3H_6-SCR, over 50% NO_x conversion can be achieved over Al_2O_3 supported catalysts at 300 °C [1–6]. Oxidation of NO to NO_2 was considered to be an essential intermediate step in the SCR reaction both with NH_3 and with hydrocarbons as reducing additives [7–10].

To achieve effective activities at lower than 200 °C, zeolite catalysts are found to be a promising choice in combination with non-thermal plasma without considering SO_2 poisoning and H_2O stability. Tonkyn et al. [11,12] reported that 50% NO_x conversion was obtained over a broad temperature range (175 °C–400 °C) by using a NTP-assisted C_3H_6-SCR on a NaY zeolite. Kwak et al. studied NTP-assisted C_3H_6-SCR over BaY and FAU zeolites and achieved best activities of over 50% from 170–290 °C [13,14]. However, most of the zeolite catalysts lost their activities above 300 °C, which leads to using a combination of zeolite and oxide catalysts in NTP-assisted SCR. Rappé et al. combined BaY and Ag/Al_2O_3 in series as a catalyst system and obtained NO_x conversion of 80% at 200 °C, 95% at 350 °C and 500 °C under steady operation [15–17].

It is interesting and valuable to find out why the SCR reactions are different over Y type zeolite and metal oxide catalysts under similar conditions. In this work, we compared the NTP-assisted C_3H_6-SCR reactions over Ag^+ exchanged ultra-stable Y zeolite (Ag/USY) and Ag^+ doped alumina (Ag/Al_2O_3) catalysts. Choosing USY zeolites is due to the better resistance of H_2O and SO_2 than common Y type zeolites. These two catalysts showed distinctive behaviors in NTP-assisted C_3H_6-SCR. In situ diffuse reflectance FT-IR spectroscopy studies showed that the promoting effect of NTP assistance was resulted from the enrichment of key SCR intermediates NCO, CN, oxygenates and some N-containing organic species.

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However, the distinction of SCR reactions over Ag/USY and Ag/Al_2O_3 at low temperatures might be attributed to different properties of the adsorbed NO_x species, as supported by the TPD profiles of these two catalysts.

2. Experimental

2.1. Catalyst preparation

The USY-based catalysts were prepared by an aqueous ion-exchange method from a USY zeolite with a Si/Al 5.2 obtained from HuaHua Co. Ltd. Before use, USY was calcined at 500 °C and washed in 0.1 M $NaNO_3$ solution for 6 h at 60 °C. After washing and drying, USY was then added to 0.01 M $AgNO_3$ with constant stirring for 24 h. The sample was collected after the resulting suspension was filtered, washed with de-ionized water, dried at 110 °C for 12 h, and calcined at 400 °C for 12 h. $Ag/\gamma\text{-}Al_2O_3$ was prepared by a single step sol–gel method as described in the literatures [18]. The Ag loadings of the catalysts used were about 5 wt.%. The physical properties of the catalysts were summarized in Table 1.

Table 1
Physical properties of 5 wt.% Ag supported on USY and $\gamma\text{-}Al_2O_3$

Samples	Calcination temperature (K)	BET surface area (m^2/g)	Pore diameter (nm)	Pore volume (cm^3/g)
Ag/USY	673	521	2.73	0.365
Ag/Al_2O_3	823	233	4.35	0.257

2.2. Activity measurement

The experimental system was of the PPCR (post-plasma catalytic reactor) type. Non-thermal plasma was generated in a cylinder-type dielectric barrier discharge (DBD) reactor with sinusoidal 5 kV high voltage of 50 Hz power input. The catalysts were placed in a quartz tube reactor with an internal diameter of 6 mm downstream from the plasma reactor. The reaction gas contains 1000 ppm NO, 1000 ppm C_3H_6, 8% O_2 and N_2 as the balance gas. The space velocity in the activity tests was fixed at about 14,400 h^{-1}. The concentrations of NO_x were measured by an $NO–NO_2–NO_x$ analyzer (Thermo Electron Model 44). Propene and byproducts after the reaction were examined online through a gas-phase chromatograph (Shimadzu GC 17A) and mass spectrograph (Omni Star).

2.3. Catalyst characterization

Temperature programmed desorption (TPD) experiments of NO_x were carried out by using 100 mg catalyst sample. Prior to the NO TPD experiment, the catalyst sample was pre-treated in a flow of 8% O_2, N_2 at 500 °C for 1 h and then was cooled down to room temperature. Chemisorption of NO was performed by passing a flow of 1000 ppm NO and 8% O_2 diluted in N_2 through the sample bed at room temperature for 1 h. After the sample was purged with N_2 until no NO concentration was detected, the TPD measurements were carried out up to 500 °C with a heating rate of 10 K min^{-1} in flowing N_2. The gas flow rate was fixed at 100 cm^3 min^{-1}.

The diffuse reflectance FT-IR (DRIFTS) measurements were carried out in situ in a high-temperature cell fitted with ZnSe windows. The samples were finely ground, placed directly to a ceramic crucible and manually pressed. The feed gas streamed into the cell at a total flow rate of 100 mL/min with or without non-thermal plasma pre-treatment. The temperature in the cell could be programmed from 20 to 800 °C. Prior to analysis, all the zeolite samples were pre-treated at 550 °C and Ag/Al_2O_3 at 600 °C in a mixture of N_2 and O_2 for 60 min, then in N_2 for 30 min to remove surface residues. Background spectra were collected after dwelling for 30 min at a given temperature, except when specially noted. One hundred scans were recorded, at a resolution of 4 cm^{-1}.

3. Results and discussion

3.1. Activities measurement

Although Ag/USY and Ag/Al_2O_3 had the same active component of Ag, the different supports led to quite different behaviors of these two catalysts in thermal C_3H_6-SCR and NTP-assisted C_3H_6-SCR. Fig. 1 shows the activities in thermal C_3H_6-SCR and NTP-assisted C_3H_6-SCR over Ag/USY and Ag/Al_2O_3, respectively. The thermal SCR over Ag/USY was almost inactive with a NO_x conversion below 15% from 80 °C to 300 °C. For NTP-assisted SCR over Ag/USY, NO_x conversions was enhanced to about 50% at low temperature of 150 °C, while it decreased to 25% at temperatures above 200 °C. From the conversions of propene, it could be seen that the difference between NTP-assisted SCR and thermal SCR resulted from the activation of the reductant. We have studied several kinds of metal oxide catalysts including Ag/Al_2O_3 and found that their activities in C_3H_6-SCR were remarkably enhanced by the assistance of NTP at 300 °C–400 °C, which was also resulted from the activation of propene reductant. It can be seen in Fig. 1 that more of 20% C_3H_6 conversion and 40% NO_x conversion were obtained with the assistance of NTP at 300 °C–400 °C. However, the activities below 250 °C were still very low and the SCR reactions nearby 150 °C were almost inactive which was quite different from those over Ag/USY.

3.2. Reaction process

3.2.1. Ag/Al_2O_3

Fig. 2a shows the dynamic change of species during NTP-assisted SCR at various reaction temperatures over Ag/Al_2O_3. It is seen that under low temperatures (i.e. 50 °C), the key intermediate of HC-SCR, –NCO (2258 cm^{-1}) specie [19–24], was obtained. Strong bands for acetate (1456 cm^{-1}), formate (1394 cm^{-1}) and N-containing organics (1710 cm^{-1} and 1645 cm^{-1}) [19–24] were simultaneously observed, which were much stronger than those found in SCR reactions under similar conditions (Fig. 2b). In the temperature range of 50 °C–250 °C, the peak intensity of bands for N-containing organics decreased, while NCO, formate, and acetate increased. It has

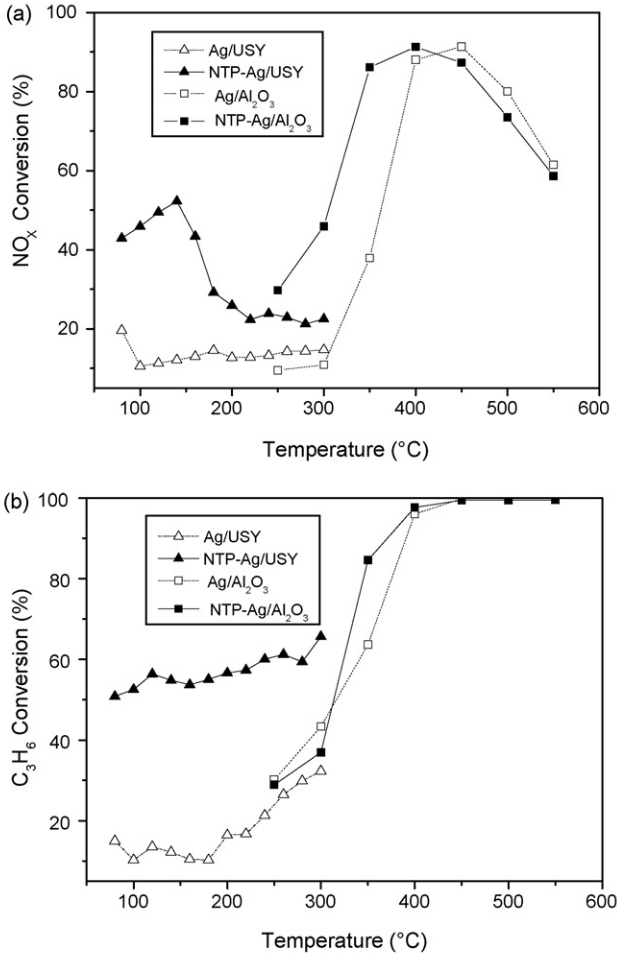

Fig. 1. Conversion of NO_x (a) and C_3H_6 (b) over Ag/USY and Ag/Al_2O_3 in thermal C_3H_6-SCR and NTP-assisted C_3H_6-SCR. Conditions: 1000 ppm NO, 1000 ppm C_3H_6, 8% O_2, N_2 as the balance gas.

been proved that N-containing organics such as R-NO_2 are active over Ag/Al_2O_3 and turn into NCO groups when reaction temperatures are appropriate. The observed increase of NCO species should result from conversion of N-containing organics. Regarding studies on the mechanism of C_3H_6-SCR over Ag/Al_2O_3 in Refs. [20–25], the enrichment of key SCR intermediates such as NCO, CN, oxygenates and other kinds of N-containing organic species would lead to the enhancement of SCR activities. In NTP-assisted C_3H_6-SCR, the IR peaks for CO_2 (2361 cm^{-1} and 2331 cm^{-1}) [20] could be observed at 250 °C, which is an indirect evidence for NO_x reduction and could be observed only above 400 °C in thermal C_3H_6-SCR. We have carried out many other experiments on NTP-assisted C_3H_6-SCR and the results showed that the reaction process above 250 °C could be explained by the typical mechanism in current literatures [19–25]. However, it was quite interesting that although the key intermediate species (e.g. NCO) were also apparently enriched by the assistance of NTP below 250 °C, the SCR reactions were still almost inactive.

3.2.2. Ag/USY

Acetaldehyde was one of the main products when propene was activated in the reaction gas of C_3H_6-SCR by NTP.

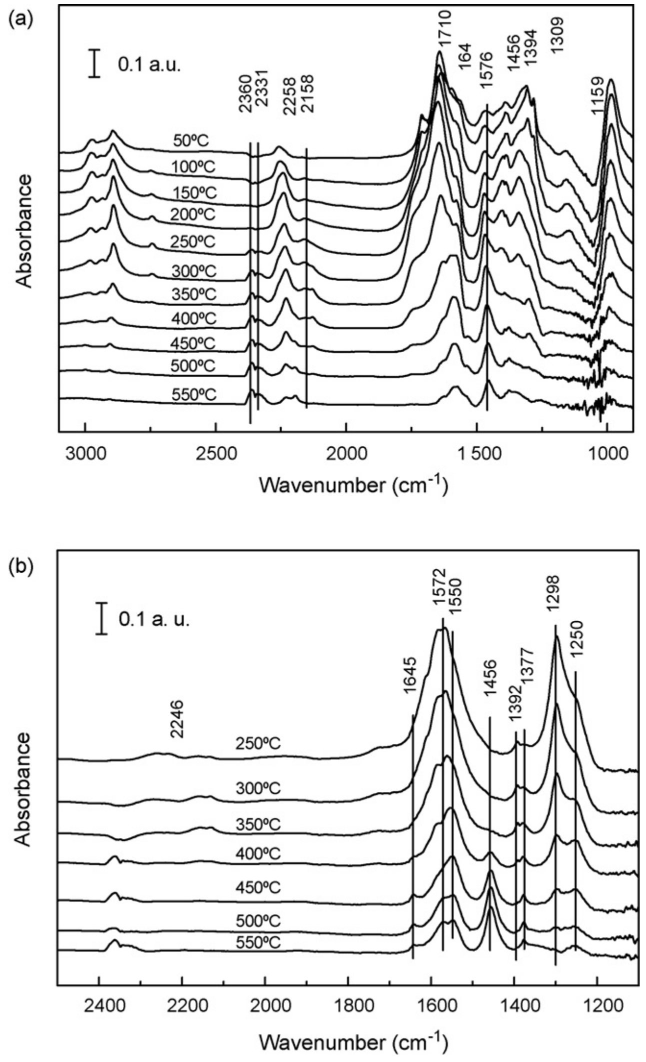

Fig. 2. DRIFTS spectra over Ag/Al_2O_3 in NTP-assisted C_3H_6-SCR (a) and thermal C_3H_6-SCR (b) at various reaction temperatures. Conditions: 1000 ppm C_3H_6, 1000 ppm NO, 8% O_2, N_2 as the balance gas.

Schmieg et al. [26] studied acetaldehyde-SCR over a NaY zeolite catalyst and obtained about 70% NO_x conversions in the temperature range of 200 °C–350 °C. Orland et al. [27] suggested that the NO_x conversion in NTP-assisted C_3H_6-SCR over NaY was obtained by the reactions between acetaldehyde and NO_2, which was the main product from the oxidation of C_3H_6 and NO in NTP, respectively. They investigated the reaction process of acetaldehyde and NO_2 over NaY at low temperatures using FTIR method and found that the mechanism involved was similar as that over Ag/Al_2O_3, namely acetaldehyde and nitrates started the nitration reactions to generate –NCO intermediates and then was the reduction reaction to generate N_2 over Ag and associated acid sites.

We studied the intermediates in thermal C_3H_6-SCR and NTP-assisted C_3H_6-SCR over Ag/USY using in situ DRIFTS and tried to find information about the reaction mechanism. Fig. 3 shows the variation of intermediates over Ag/USY with reaction temperature in thermal C_3H_6-SCR. It was quite different from what was observed over Ag/Al_2O_3, and strong

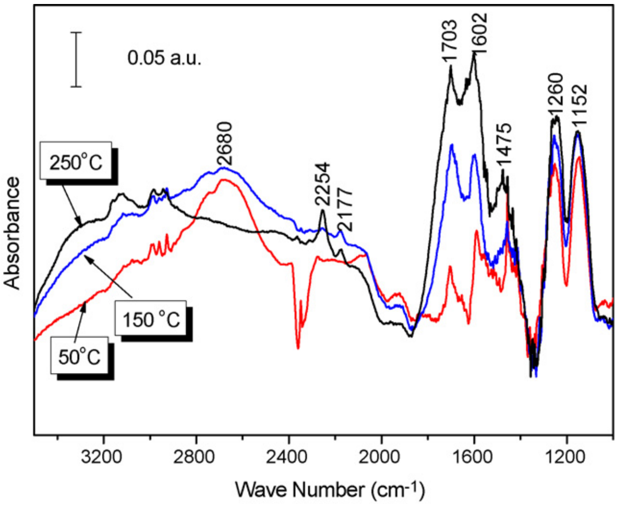

Fig. 3. Dynamic change of intermediate species in thermal C_3H_6-SCR over Ag/USY with various reaction temperatures. Conditions: 1000 ppm C_3H_6, 1000 ppm NO, 8% O_2, N_2 as the balance gas.

and wide IR peaks centered at 2680 cm^{-1} was observed over Ag/USY. Although accurate assignments for these peaks are not available, they should be of some kinds of N-containing species, which will be discussed later. The peaks at 2680 cm^{-1} remained strong and no NCO specie was observed at 50 °C and 150 °C. This suggested that the unknown N-containing species were stable at low temperatures and the SCR reactions were inactive, which was consistent with the activities observed. After the reaction temperature increased to 250 °C, obvious IR peaks for NCO and CN occurred at 2254 cm^{-1} and 2177 cm^{-1}. However, the activities of C_3H_6-SCR were still very low at the reaction temperatures around 250 °C. Some evidences on this phenomenon could also be found in the spectra. It was seen in Fig. 3 that although the IR peak intensities for NCO increased with time, the intensities of IR peaks for nitrate and nitrite species (1602 cm^{-1}, 1260 cm^{-1} and 1155 cm^{-1}) [20,28] simultaneously increased. According to the mechanism of HC-SCR over metal oxide catalysts and some zeolite catalysts (e.g. NaY), the adsorbed species of NO_x (ad-NO_x) were involved in the SCR reaction. The increase of ad-NO_x species indicated the inability of reducing NO_x to N_2. The peaks for unknown N-containing species at 2680 cm^{-1} weakened at 250 °C, which might be converted to nitrate and nitrite species and caused an increase of peak intensity for these species in the IR spectra.

Fig. 4a shows the variation of intermediates with reaction temperature in NTP-assisted C_3H_6-SCR over Ag/USY. In these spectra, we did not find so strong IR peaks at 2680 cm^{-1} as observed in thermal C_3H_6-SCR, indicating that after the reaction gas was activated by NTP, some reaction pathways were different from those of thermal C_3H_6-SCR. Although the IR peaks for nitrates, oxygenates and N-containing organic species in the region of 1720–1400 cm^{-1} was not easily distinguished, they were stronger in NTP-assisted C_3H_6-SCR than in thermal C_3H_6-SCR. As the reaction temperature increased from 50 °C to 250 °C, the bands for CO_2 and NCO steadily strengthened. However, the activities at this temperature range were of volcano shape with a peak at around 150 °C (as seen in Fig. 1). As considering the consumption of nitrate species, it was found that various kinds of nitrates demonstrated different behaviors (Fig. 4b). The IR peak intensity for bridged nitrate at 1606 cm^{-1} almost linearly increased with temperature. It seemed not to be an active participant of SCR reactions. The IR peak intensity for unidentate nitrate at 1260 cm^{-1} showed a volcano shape with a maximum at 100 °C. The consumption of unidentate nitrate species indicated that unidentate nitrate was mainly involved in the reduction process of NO_x over Ag/USY. Although the amount of unidentate nitrate increased, the reduction of NO_x still occurred according to the SCR activities below 100 °C. Since C_3H_6 could not be entirely activated in NTP, some reaction pathways that occurred in thermal C_3H_6-SCR still existed over Ag/USY in NTP-assisted C_3H_6-SCR. They might contribute to the increase of ad-NO_x as discussed above and masked the consumption of unidentate nitrate species below 100 °C.

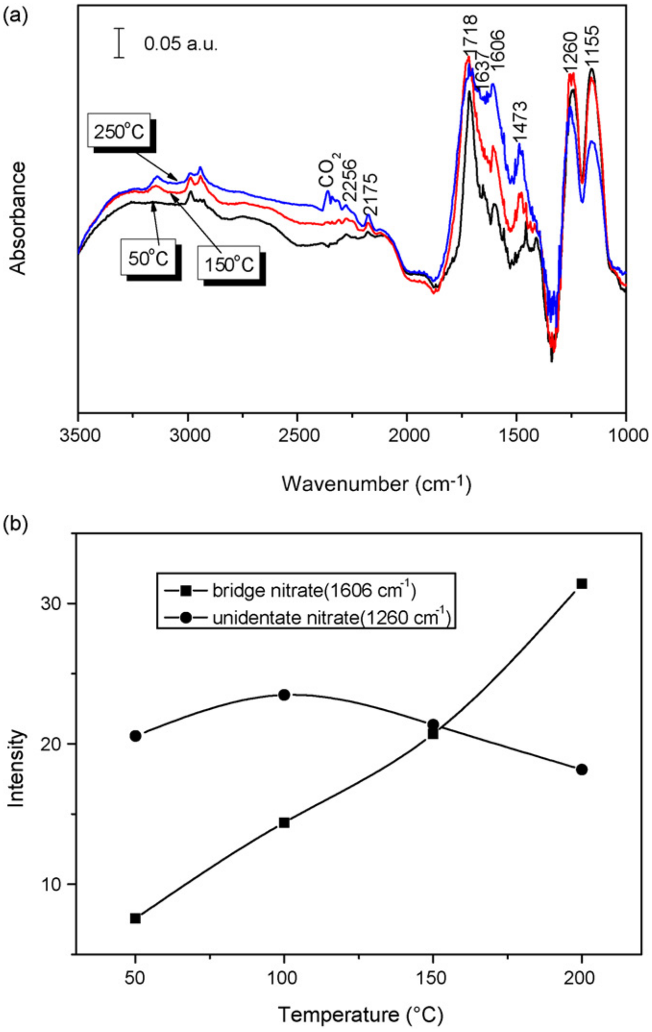

Fig. 4. DRIFTS spectra in NTP-assisted C_3H_6-SCR over Ag/USY (a) and the variation of the quantity of nitrates with reaction temperature (b). Conditions: 1000 ppm C_3H_6, 1000 ppm NO, 8% O_2, N_2 as the balance gas.

Fig. 5 demonstrates the variation of surface species during propene adsorption, thermal C_3H_6-SCR and consecutively of NTP-assisted SCR over Ag/USY at 150 °C. The adsorption of

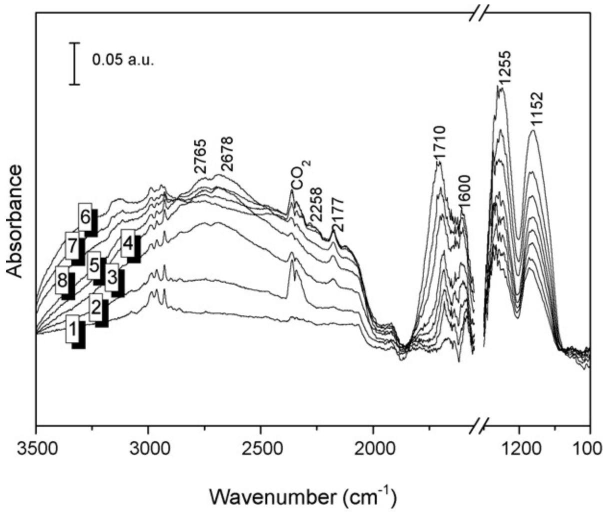

Fig. 5. DRIFTS spectra over Ag/USY at 150 °C in the following operations: $C_3H_6 + O_2$ adsorption for 30 min (1), SCR for 10 min (2), 20 min (3), 30 min (4) and 60 min (5), NTP-assisted SCR for 10 min (6), 20 min (7) and 40 min (8). Conditions: 1000 ppm C_3H_6, 1000 ppm NO, 8% O_2, N_2 as the balance gas.

$C_3H_6 + O_2$ (step 1) mainly resulted in surface acetate species (3073 cm^{-1} CH_3, 3000–2800 cm^{-1} CH and CH_2, 1572 cm^{-1} COO^-) [20] and adsorbed propene. There were no obvious IR peaks at 2680 cm^{-1} in this period. Soon after NO was added into the feed gas (step 2), strong IR peaks attributable to CO_2 (2360 cm^{-1} and 2334 cm^{-1}) were observed, which suggested that the addition of NO promoted the oxidation of surface organic species to CO_2. As the reaction process progressed (steps 3–5), IR peaks near 2680 cm^{-1} (2765 cm^{-1} and 2678 cm^{-1}) occurred and strengthened. This phenomenon suggested that the unknown species at 2680 cm^{-1} was not oxygenates from the reactions between C_3H_6 and O_2 but N-containing species from the reactions among C_3H_6, O_2 and NO_x. After NTP was applied (steps 6–8), the bands at 2765–2678 cm^{-1} continually weakened with time while new species at 2279–2256 cm^{-1} and 3150–3100 cm^{-1} strengthened. The bands occurring near 2231 cm^{-1} were assigned to –NCO groups. It is proposed that the reactions over Ag/USY turn to a typical NCO leading SCR pathway by application of non-thermal plasma.

3.2.3. Similarity and difference

In a PPCR system used in this paper, the activity enhancements in NTP assisted C_3H_6-SCR were resulted from the activation of reaction gas by NTP no matter over Ag/USY or Ag/Al_2O_3. Although the pathways in thermal C_3H_6-SCR over Ag/USY were different from those over Ag/Al_2O_3, the influences of NTP assistance to these two catalysts were similar in some ways. The mechanism of NO_x reduction was similar according to our results and the studies in other literatures [22,27], namely some key intermediates such as NCO, CN, etc., were generated by various kinds of reactions and consecutive reduced NO_x or ad-NO_x. The assistance of NTP enriched NCO, CN, oxygenates and some N-containing species over Ag/USY and Ag/Al_2O_3. These intermediates led to active SCR reactions over Ag/USY below 250 °C, which, however, did not show obvious promotion for SCR reactions over Ag/Al_2O_3 at low temperatures. No evidences were found to prove that different supports changed the behaviors of NCO, CN and oxygenate species. However, some information suggested that the properties of adsorbed NO_x species might influence the SCR process and were related to the catalyst support. Shimizu et al. [29] suggested that the stable nitrates might occupy the active sites over Ag/Al_2O_3 and were unfavorable for the SCR reactions at low temperatures. The properties of ad-NO_x species were found to be important for SCR reactions over Ag/USY, which were probably essential over Ag/Al_2O_3. The difference in the ad-NO_x species over these two catalysts were examined in next section.

3.3. Behaviors in NO_x adsorption

Fig. 6a shows the TPD profiles of NO + O_2 adsorption over Ag/Al_2O_3. Two NO adsorption peaks were observed at 110 °C and 480 °C, which were apparently strong than those could be observed over Ag/USY (Fig. 6b). It had been revealed in many literatures that the nitrates over Ag/Al_2O_3 were quite stable at low temperatures. Only one NO_2 adsorption peak was found at about 410 °C attributable to nitrates. Fig. 6b shows NO + O_2 TPD profiles over Ag/USY. The adsorption of NO was very

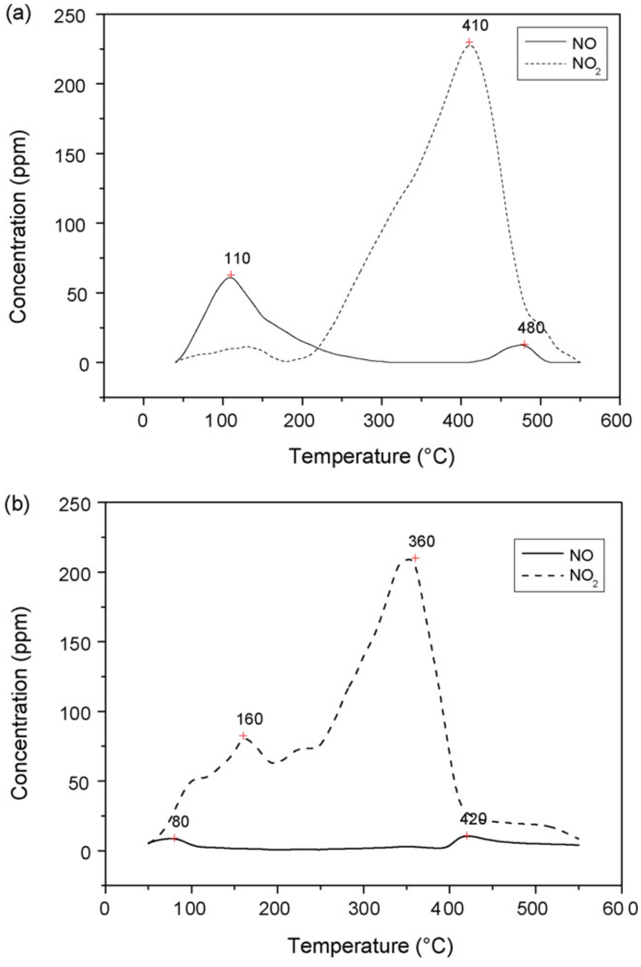

Fig. 6. TPD profiles of NO and NO_2 on Ag/Al_2O_3 (a) and Ag/USY (b).

weak with two peaks located at 80 °C and 420 °C. However, one more peaks nearby 160 °C was observed in the NO_2 TPD profile of Ag/USY as compared with Ag/Al_2O_3. These adsorption peaks should have resulted from the surface nitrates formed on the Ag or Al sites over Ag/USY. From the TPD profiles above, we would see that the nitrate species were more unstable over Ag/USY than over Ag/Al_2O_3. If stable nitrates occupied the active sites over Ag/Al_2O_3 and were unfavorable to SCR reactions at low temperatures as suggested by Shimizu et al. [29], unstable nitrates over Ag/USY would result in more effective SCR reactions, which was proved by the activity of these two catalysts in NTP-assisted C_3H_6-SCR.

Fig. 7a shows the ad-NO_x species over Ag/γ-Al_2O_3 during NO + O_2 adsorption with or without NTP pre-treatment. Typical nitrate and nitrite absorbance bands were observed: unidentate (1550 cm^{-1} and 1232 cm^{-1}), bidentate (1590 cm^{-1} and 1315 cm^{-1}), bridging (1612 cm^{-1}) nitrates, and nitrite (1473 cm^{-1}) species [20,26,28]. The application of NTP increased the intensity of IR peaks for bidentate nitrate (1315 cm^{-1}) and nitrite (1473 cm^{-1}) species but decreased those for bridge and unidentate nitrate species (1600–1500 cm^{-1}). Bidentate nitrate was very stable even at high temperatures (e.g. 500 °C). The application of NTP probably promoted the formation of more stable ad-NO_x species. Fig. 7b shows the ad-NO_x species over Ag/USY during NO + O_2 adsorption with or without NTP pretreatment. IR peaks at 1926 cm^{-1}, 1625 cm^{-1}, 1596 cm^{-1}, 1263 cm^{-1} and 1173 cm^{-1} were attributed to various kinds of ad-NO_x species. The pre-treatment of plasma on Ag/USY mainly enhanced the quantity of nitrates at 1625 cm^{-1} and 1596 cm^{-1}. The application of NTP also increased the amounts of bidentate nitrate species (1596 cm^{-1}) but did not decrease the amount of ad-NO_x species at 1263 cm^{-1} that was active participants in SCR reactions.

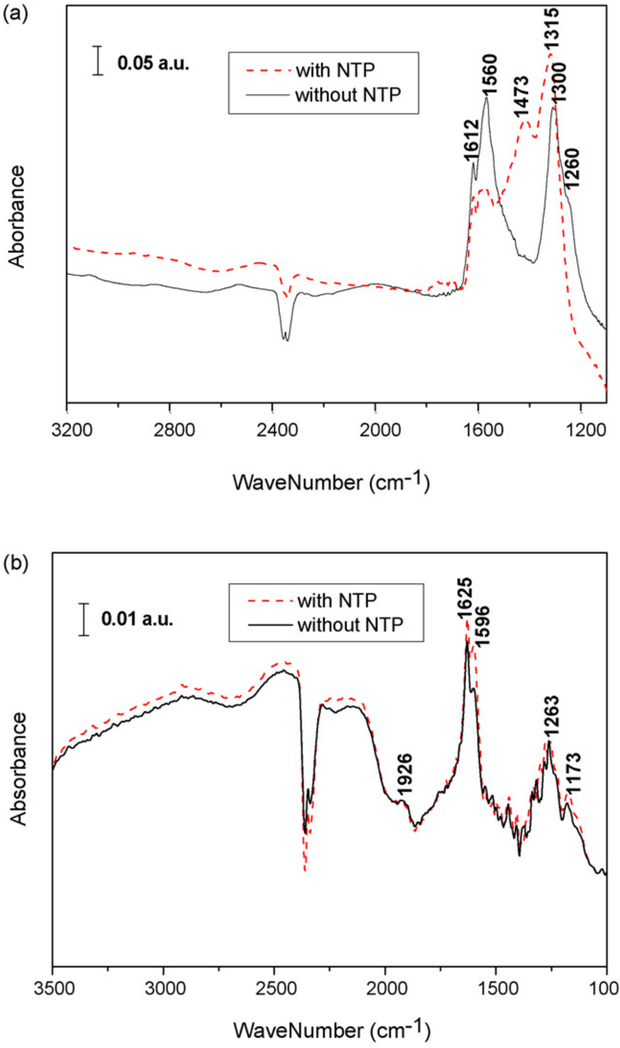

Fig. 7. DRIFTS spectra during NO + O_2 adsorption over Ag/Al_2O_3 (a) and Ag/USY (b) with or without NTP pretreatment. Conditions: NO 1000 ppm, 8% O_2, N_2 as the balance gas.

4. Conclusions

With the assistance of NTP, the activities of C_3H_6-SCR were promoted over Ag/γ-Al_2O_3 and Ag/USY. However, these two kinds of catalysts showed distinctive behaviors in different temperature region. NTP assistance showed obvious promotional effect on Ag/Al_2O_3 in the temperature range of 300 °C–400 °C while on Ag/USY was in the region of 100 °C–200 °C. After the application of NTP, key SCR intermediates such as NCO, CN, oxygenates and other N-containing organic species were apparently enriched, which resulted in the promotion of SCR activity. However, the SCR reactions over Ag/Al_2O_3 were inactive below 250 °C in presence of abundant key SCR intermediates. DRIFTS studies on NTP-assisted C_3H_6-SCR over Ag/USY showed that these reactions were effective near 150 °C. Bridge nitrate seemed not to be an active participant in the SCR reactions whereas unidentate nitrate did.

Differences in the behaviors of NCO, CN and oxygenate species were not found in NTP-assisted C_3H_6-SCR over Ag/USY and Ag/Al_2O_3. However, the variation of the properties of adsorbed NO_x species between different catalysts might be responsible for the distinct behaviors of Ag/USY and Ag/Al_2O_3 in NTP-assisted C_3H_6-SCR. The nitrate species were more stable over Ag/Al_2O_3 than over Ag/USY at low temperatures. Stable nitrates might occupy the active sites over the catalysts and were unfavorable to SCR reactions, namely, appropriate stability of nitrates might be important for effective SCR reactions at low temperatures.

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